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THERMAL DECOMPOSITION STUDIES OF NITROCELLULOSE BY AN ACCELERATING RATE CALORIMETER

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THERMAL DECOMPOSITION STUDIES OF NITROCELLULOSE BY AN ACCELERATING RATE CALORIMETER

by

P. Lee* and R. Vandebeek**

ABSTRACT

Thermal decomposition of nitrocellulose (NC) has been studied with an accelerating rate calorimeter (ARC) under various sample/bomb systems, heating parameters and different atmospheres. The self-heat curve shows the exothermic reaction and the time-temperature-rate-pressure data are recorded at the sensitivity of 0.02°C/min.

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INTRODUCTION

When a reactive material is stored at an elevated temperature, decomposition can occur at low rates. Its temperature rise is highly dependent on the heat loss to the surrounding medium. As temperatures increase, the decomposition rates increase. The stored material system enters a critical state when the heat generation rate exceeds the rate of heat dissipation.

The evaluation of thermal hazards associated with the manufacture, transport and storage of explosives and propellants is a new research project at the Canadian Explosives Research Laboratory (CERL). The thermal analyzers, e.g. DSC, DTA, TG as well as MVP test (15), Abel test (15) and stabilizer depletion test (13) are commonly used for thermal stability evaluation of explosives. However, there are limitations for each method; for example, the composition of a sample of a few milligrams taken from a grossly heterogenous suspension is not necessarily representative of the whole mass in microanalytical techniques.

Accelerating rate calorimeter (ARC) is a new analyzer for hazard evaluation of exothermic reactive chemicals. Its primary function is to maintain a sample in an adiabatic state and permit it to undergo thermal decomposition due to self-heating. Once self-heating is detected as evidenced by a slow steady temperature increase, the sample is left alone to complete its thermal spiral. The ARC is used for explosives thermal stability studies to provide the data of exothermic reactions and evaluate thermal chemical hazards.

EXPERIMENTAL

The details of the design, operation and performance of the ARC have been published in other literature (1,2,3,5,6,7). A brief discussion of the instrumental design and the operational logic follow. The operation of the ARC involves a sample in a bomb where the bomb plus contents is maintained adiabatically with respect to its environment. In order to achieve an adiabatic condition over a temperature range of ambient to 500°C and temperature rates of 0.02°C/min to 10°C/min, the ARC uses a digitally controlled thermocouple/heater feedback system (4). Figure 1 illustrates the calorimeter part of the ARC. The jacket is composed of three zones, top, side and base which

are individually heated and controlled by Nisil/Nicrosil (type N) thermocouples. The thermocouples are cemented on the inside surface of the jacket and the same type of thermocouple is clamped directly on the outside surface of the bomb. All the thermocouples are referenced to the ice point which is designed to be stable to within 0.01°C. The placement of the bomb thermocouple has to be repeatable in every experiment. In normal operation, without extreme care, the thermal contact is not very reproducible which will cause highly undesirable random scattering of the calibration curves and drifting problems during the run. The drift corrections are performed normally every 50°C for the entire temperature range by applying the required offset voltage. The pressure of the reaction system is monitored with a 0-2500 psi pressure transducer. For safety precautions, the entire calorimeter is mounted inside a containment vessel.

A search for a reaction exotherm is accomplished by elevating the sample temperature by a fixed increment (step heat) and then checking to see if the sample self-heat rate exceeds a user-selected threshold, usually 0.02°C/min. Once an exotherm is detected, automatic collection of time, temperature, rate and pressure data is carried out until the reaction has finished and the self-heat rate has dropped back below the threshold value. Figure 2 illustrates a typical run sequence for detecting and following an exothermic runaway reaction.

In the experiments with the ARC, part of the heat generated by the exotherm is used in raising the temperature of the bomb and this aspect must be taken into account. The thermal inertia factor ϕ is a critical factor in the analysis where data must be corrected to allow for the effect of the test bomb heat capacity on the response of the system. In an ideal case, the ϕ factor is equal to 1. Thermal inertia is defined as,

$$\phi = 1 + \frac{C_v \text{ (bomb) } M \text{ (bomb)}}{C_v \text{ (sample) } M \text{ (sample)}}$$

where C_v is the heat capacity and M is the mass. There are several types of bombs which can be used depending on the materials being tested. The light weight titanium bomb is used when low thermal inertia is desired and the Hastelloy C cylindrical bomb can be used for high melting point or viscous samples.

RESULTS AND DISCUSSIONS

Nitrocellulose (NC) from the Propellant Department, PCV (17), laboratory sample, NC wet, reference No. 1290 (Oct. 2, 1979) was used for thermal stability studies. The sample/bomb system response, effect of thermal inertia, effect of heating parameters and decomposition under different atmospheres were studied.

I. HASTELLOY C CYLINDRICAL BOMB (approximately 50 gm)

The Hastelloy C bomb was used for the first few runs through the ARC. Since the NC ignited vigorously upon heating, only 0.25 gm sample could be used. This resulted in a high thermal inertia factor (ϕ) which slowed down the reaction, so that the lighter bomb was used for further studies.

II. TITANIUM 0.81 mm (0.032") WALL SPHERICAL BOMB (approximately 7 gm)

The Ti bomb, having an opening at the top, was sealed with self-adhesive Teflon tape. This bomb was used for the following studies.

A. Effect of Thermal Inertia (ϕ)

The step heat logic mentioned above was employed for the thermal inertia study. A series of experiments were done by varying the weight of the desiccator dried NC so as to vary the ϕ factor. The run started at 130°C and ended at 180°C with a heat step of 2°C. Since the ignition of the larger sample could cause damage to the calorimeter, the run was terminated when a 0.5°C/min temperature rate was detected. The 0.5°C/min rate was chosen because enough data was available for the self-heat curve and the time was long enough for cooling the sample with compressed air before ignition occurred. The results showed that the exotherm onset was detected at a lower temperature with a lower ϕ factor. The lowest temperature detected for the exotherm occurrence was 136.5°C at 0.02°C/min with a ϕ factor of 6.4. No ignition occured with ϕ factor equal to 80.4 which is shown in Fig. 4. In some cases, there was a temperature drift problem. The stability of the heat step was indicated by temperature rate. In Fig. 4, Run No. 17 and 19 with negative drift at 0.02°C/min and 0.05°C/min respectively were compared with duplicate runs, Run No. 29 and 20. Both curves showed that the exotherms were shifted to higher temperature. Figure 5 is the plot of sample weight vs ini-

tial temperature of NC decomposition and the results are summarized in Table 1. Figure 5A and 5B show the infrared absorption spectra which indicate no change in NC after heat up to 0.5°C/min. Two absorption bands at 1667 and 1282 cm^{-1} in the infrared spectrum characterize the $O-NO_2$ group.

B. Temperature Effect on NC Storage

Nitrocellulose was kept in a 75°C oven and samples were removed for the ARC step heat test after various lengths of storage time; 61 days, 68 days, 83 days, 101 days, 111 days and 141 days. One quarter gram samples with ϕ factors about 20 were used in the test so that vigorous ignition and damage could be avoided. All the samples ignited at step heat with similar self-heat curves and no significant change could be detected up to 141 days aging. Figure 5A and 5C compare the infrared spectra of NC which indicated no change after 141 days aging. Per cent total nitrogen of NC was analyzed by Perkin-Elmer C,H,N, elemental analyzer model 240 and a titrimetric method, trans-nitration involving reduction with Ti (III) (14). The results of per cent nitrogen by elemental analyzer were consistently lower than the titrimetric method probably due to the fast ignition which caused incomplete combustion of the NC sample. However, both methods indicated no significant change of per cent nitrogen in NC after 141 days storage at 75°C. Table 2 compares the analytical results of NC with and without heating history.

Table 1 - Effect of thermal inertia on the initial temperature of NC decomposition

ARC Run No.	Wt. of NC (gm)	ϕ factor	Initial temp T_o (°C)	Initial rate M_o (°C/min)	Temperature (°C) at *0.02°C/min	0.05°C/min	0.5°C/min	Temperature (°C) before ignition	Rate (°C/min) before ignition
20	0.0622	80.4	155.69	0.027	155.0	157.3	-	-	-
17	0.1212	43.3	155.06	0.063	152.0	154.0	165.0	175.58	1.576
29	0.1213	43.2	148.74	0.020	149.0	152.3	163.0	171.28	1.205
12	0.2504	19.9	148.00	0.022	147.5	150.5	159.5	162.15	1.125
34	0.2511	21.3	146.93	0.028	147.0	148.9	159.5	164.54	1.386
28	0.3512	15.6	144.90	0.026	144.5	146.9	157.5	162.06	1.355
14	0.4163	11.9	138.13	0.021	138.0	143.1	156.5	160.74	1.551
23	0.4359	12.0	142.57	0.028	140.0	144.6	156.5	160.13	1.212
45	0.5500	10.4	140.90	0.030	139.5	143.0	155.0	-	-
40	0.6000	9.5	140.86	0.027	140.0	142.3	155.0	-	-
41	0.7500	7.8	140.85	0.034	138.0	141.9	154.0	-	-
47	0.8000	7.4	138.84	0.022	138.0	141.4	153.0	-	-
49	0.9000	6.7	138.84	0.026	138.0	141.4	153.5	-	-
48	0.9500	6.4	136.78	0.021	136.5	139.8	151.0	-	-

* Temperature (°C) at 0.02°C/min is the extrapolated value

Table 2 - Comparison of analytical results of NC with and without heating history

ARC Run No.	In 75°C oven (days)	% nitrogen in NC		ARC data				
		elemental analyzer	titrimetric method	ϕ	Initial temp T_o (°C)	Initial rate M_o (°C/min)	Temperature (°C) before ignition	Rate (°C/min) before ignition
	*	9.38						
12	0	9.38	13.0	19.9	148.00	0.022	162.15	1.125
34	0	9.38	13.0	21.3	146.93	0.028	164.54	1.386
24	61	-	-	20.9	144.41	0.028	160.96	0.891
31	68	9.15	13.2	21.2	147.14	0.029	164.18	1.290
32	83	8.43	12.5	21.2	140.63	0.019	166.30	1.675
	*	11.52						
37	101	10.35	13.0	21.3	146.94	0.025	161.47	1.050
43	111	10.50	13.0	21.3	146.90	0.030	163.93	1.366
	*	12.35						
59	141	12.33	12.9	21.3	144.82	0.027	162.36	1.024

*A control for % nitrogen determination by elemental analyzer, desiccator dried NC without heating history

C. Effect of Isothermal Age Time at Constant Temperature

The decomposition reaction of nitrocellulose brought about by heating is an autocatalytic process and is a complex reaction (15, 16). For autocatalytic systems where the chemical agent responsible for the catalysis could accumulate with time under isothermal conditions, the reaction is chemically accelerated beyond that expected by thermal effects.

In this study, 0.25 gm desiccator dried NC with a ϕ factor of about 20 was isothermally aged at 130°C for a pre-determined length of time; 24 hours and 72 hours. Then it was step heated 2°C to generate the self-heat curve. The results showed NC was stable for 72 hours at an isothermal age temperature of 130°C. An autocatalytic reaction was hardly detected by comparing the runs. Since the bomb was only enclosed with self-adhesive Teflon tape, it was possible that the evolved gas escaped and there was no accumulation of catalyst.

D. Effect of Isothermal Age Temperature at Constant Time

One quarter gram NC samples were isothermally aged at 130.0°C, 133.0°C, 134.0°C, 134.5°C and 135.0°C for 72 hours. After the isothermal period, the samples were step heated at 2°C intervals to generate the self-heat curve. NC was stable at 130.0°C, 134.0°C and 134.5°C for more than 72 hours. After step heat, ignition occurred for the 130.0°C run but not for 134.0°C and 134.5°C runs. The per cent nitrogen in residues from the 134.0°C and 134.5°C runs determined by elemental analyzer was 4 to 5 times higher than by titrimetric method. It could be explained by the fact that the elemental analyzer gives the total nitrogen in the NC residue while the titrimetric method is applicable only to the determination of inorganic and organic nitrate salts and nitramines. The low percentage nitrogen by titrimetric method indicated that the nitrate group in the NC residue was altered to another form upon heating above 130°C. The 133.0°C isothermal age study showed consistent initial temperature of the exotherm but not the time to the initial temperature. The 135°C isothermal age runs showed exotherms starting at about 600 minutes with initial temperatures of 140°C. Table 3 summarizes the NC decomposition results of various isothermally aged temperatures for 72 hours.

Table 3 - Results of NC decomposition at various isothermal temperatures for 72 hour aging studies

ARC Run No.	Isothermal age temperature (°C)	Initial temp T_o (°C)	Initial rate M_o (°C/min)	Time to T_o (min)	Temperature (°C) before ignition	Rate (°C/min) before ignition	% nitrogen elemental analyzer	% nitrogen titration
16	130.0	147.05	0.049	4485	161.18	1.640	-	-
42	133.0	139.65	0.020	2023	163.79	1.771	-	-
46	133.0	140.60	0.020	1010	158.77	1.050	-	-
*							9.38	13.0
21	134.0	144.57	0.030	4417	-	-	4.62	1.3
22	134.0	145.27	0.042	4444	-	-	4.75	1.1
*							11.52	-
36	134.5	151.49	0.053	4457	-	-	5.53	1.1
35	135.0	140.13	0.018	622	160.13	1.135	-	-
39	135.0	140.93	0.019	693	162.95	1.720	-	-

*A control for % nitrogen determination by elemental analyzer, desiccator dried NC without heating history

III. TITANIUM 0.81 mm (0.032") WALL SPHERICAL BOMB (approximately 8 gm)

Ti spherical bomb was used in the following NC decomposition studies. The sample loaded bomb was connected to the pressure transducer equipped with a pressure limiting valve for pressure measurement. It was difficult to obtain reproducible pressure data due to the particles produced during the ignition blocking the tubing connecting the bomb to the pressure transducer. It was necessary to clean the pressure transducer every two or three runs.

A. Original Calorimeter Design

Figure 6A demonstrates the design of the original calorimeter top which was used in the closed system studies in air atmosphere. One quarter gram NC was step heated 2°C to generate the self-heat curve. Time-temperature-ratepressure data could be recorded automatically whenever the exotherm occurred. Step heat studies with a ϕ factor of 20 indicated that the NC decomposition temperature occurred in the range of 132°C to 136°C which was more than 10°C lower than the open bomb system. Results are summarized in Table 4.

No successful isothermal studies were achieved using the closed bomb system. It was impossible to obtain steady isothermal age temperatures for any length of time even at 110°C. No apparent explanation for this has been determined.

Table 4 - NC decomposition studies with closed system, original calorimeter top

ARC Run No.	Start temp (°C)	Initial temp T_o (°C)	Initial rate H_o (°C/min)	Temp (°C) at 0.05 °C/min	Temp (°C) before ignition	Rate (°C/min) before ignition	Max pressure (MPa)	(psia)
53	50	131.52	0.050	131.5	143.11	1.500	2.9	424.4
60	100	130.43	0.050	130.0	145.06	1.071	2.8	399.3
62	120	136.77	0.033	136.0	160.87	0.909	2.5	361.8
63	120	132.72	0.031	134.0	151.42	1.589	3.2	461.8
50	130	134.90	0.050	135.0	147.51	1.272	3.0	432.2

B. Modified Calorimeter Top Design

Figure 6B shows the modified calorimeter top. It was designed so that the sample could be detected in different atmospheres and any leakage in the system could be easily detected. However, this introduced several problems. The dead volume increased, the evolved gases now had two paths to follow and the extended tubing inside the bomb could partially absorb the evolved heat.

Effect of oxygen, inert gas and catalyst were studied by using the modified calorimeter top. NC runs with a ϕ factor of 20 were studied by 5°C step heat to generate self-heat curves. The results are presented in Table 5. The effect of oxygen on NC decomposition was examined by testing the sample in an air atmosphere. The exotherm temperature at a rate of 0.05 °C/min was higher with the modified system than the results obtained with the original calorimeter system. The effect of inert gas was tested after the sample/bomb system was flushed with helium. The results indicate that the initial temperature of exotherm in helium atmosphere was higher than in air atmosphere. Effect of catalyst was studied by running NC in NO_2 atmosphere. Unfortunately, no stable or reproducible results could be obtained since the heat steps were only stable at low temperatures. Various tests have been carried out to identify that the cause of instability was due to NO_2. It was done by using different thermocouples and various conditions. Figure 7 gives typical temperature-time and rate-time profiles for an ARC experiment of NC decomposition. Figure 8 is the plot of temperature (°C) vs time (minutes) in which the results are compared in He and air atmospheres.

Table 5 - NC decomposition studies with closed system, modified calorimeter top

ARC Run No.	Atmosphere	Start temp (°C)	Initial temp T_o (°C)	Initial rate M_o (°C/min)	Temp (°C) at 0.05 °C/min	Temp (°C) before ignition	Rate (°C/min) before ignition	Max pressure (MPa)	(psia)
64	Air	25	145.90	0.042	146.0	165.14	1.071	1.0	148.1
68	Air	50	141.04	0.033	142.0	163.07	1.081	1.3	182.4
69	Air	100	131.86	0.024	136.0	160.89	0.893	1.4	208.7
65	Helium	50	150.87	0.048	151.0	164.05	1.176	1.6	229.2
66	Helium	50	150.88	0.035	151.5	163.02	0.898	1.2	176.3
76	Helium	50	141.08	0.028	145.7	162.70	1.000	0.6	93.4
70	Helium	100	151.19	0.055	151.0	163.36	1.067	1.1	152.9
71	Helium	110	148.09	0.045	148.5	162.10	1.025	1.3	188.8

CONCLUSIONS

The ARC is an automated laboratory instrument which aids in experimentally determining the time, temperature and pressure relationships of any exothermal reaction in an adiabatic environment. It has a very high sensitivity of 0.02 °C/min for the detection of exothermic reactions. The data produced by the ARC could be applied to the evaluation of thermal and pressure hazard potentials of reactive materials. However, NC decomposition results from the ARC are dependent on the sample/bomb system response, the mass of the bomb, the sample contact area in the bomb and the interfacial heat transfer coefficient for the materials. Thus a question has been raised about the accuracy of the data obtained under various ARC operating conditions, especially if the material under study is solid state. Due to the limitations of the instrument, it is difficult to obtain accurate thermal kinetic parameters at the present stage.

The initial temperature of the exotherm for NC decomposition in air atmosphere was detected in the range of 131°C to 148°C depending on the experimental system used. These data indicate that the decomposition of NC starts at a lower temperature using the ARC than the other thermal analyzers which detect the exothermic peaks at over 200°C (8-12). Nitrocellulose shows no change after 141 days aging at 75°C. The use of accelerating rate calorimetry for the thermal stability evaluation of NC has proved useful. Additional work on NC propellants to determine the effects of stabilizers and deterrent coatings on thermal stability is planned.

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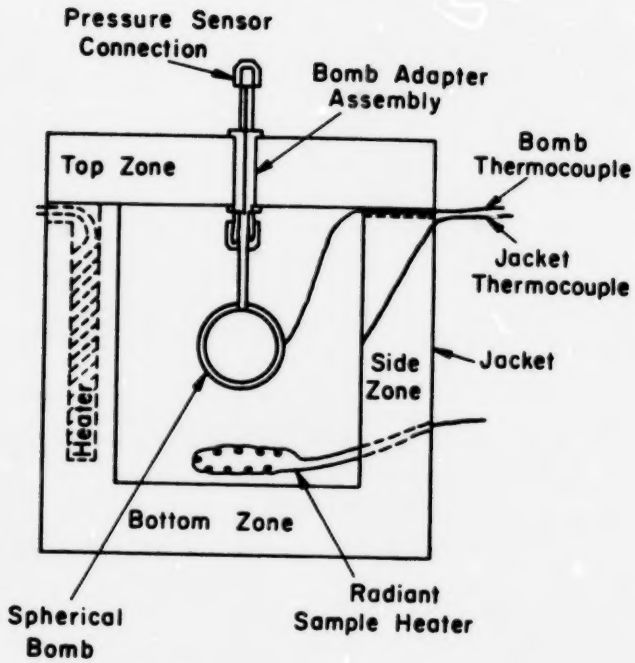

Fig. 1 - Calorimeter assembly and sample system

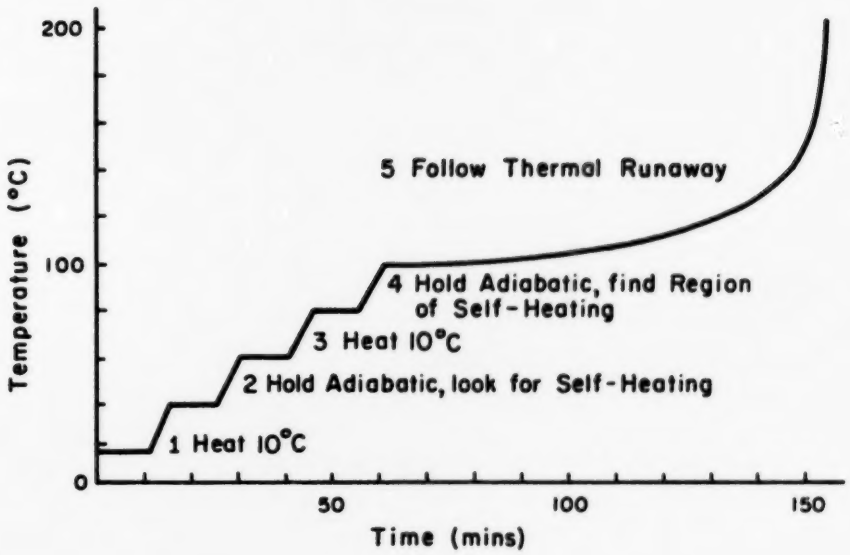

Fig. 2 - Heat-wait-search logic operation

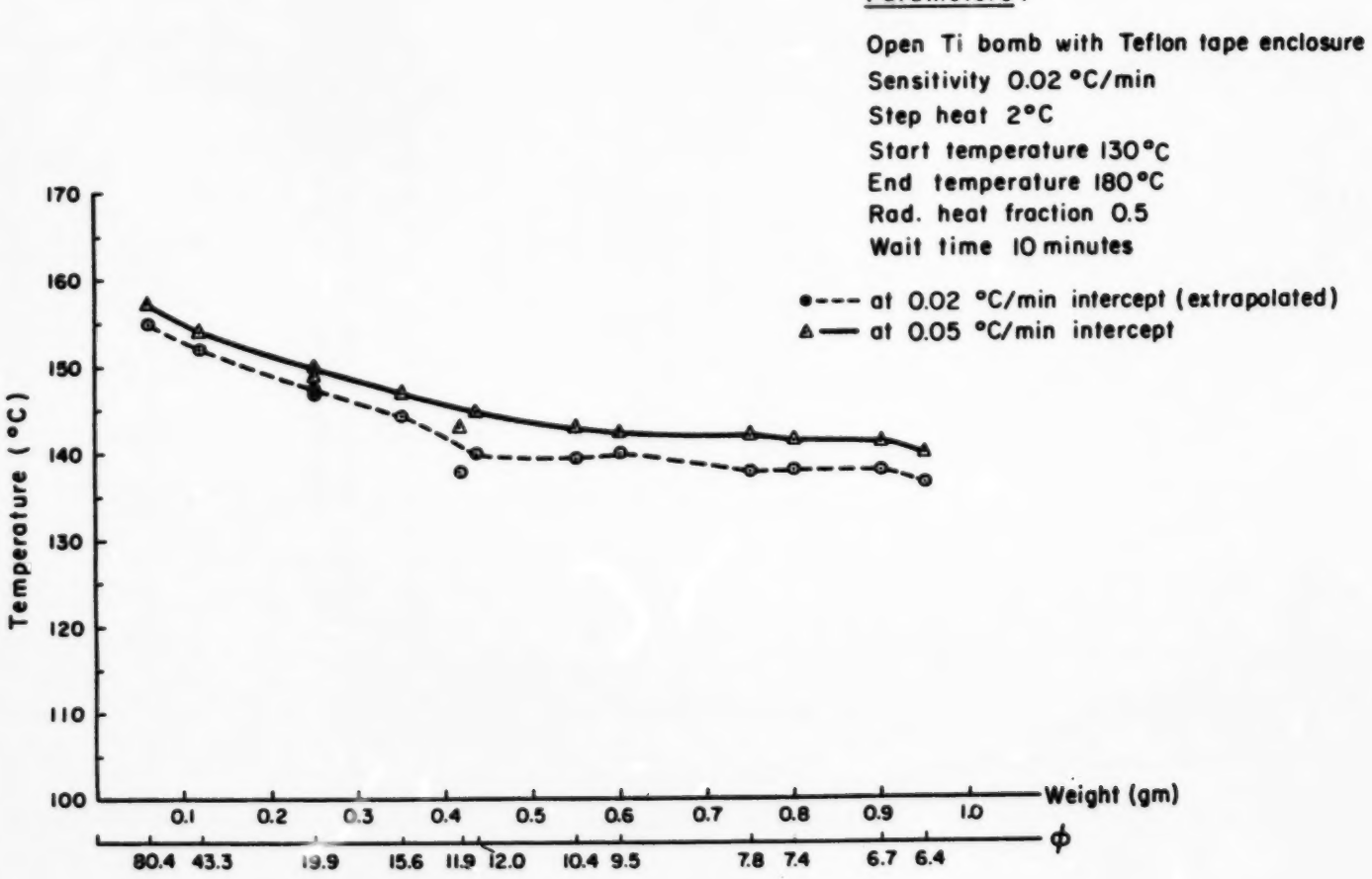

Fig. 3 - Effect of thermal inertia (ϕ) on the initial temperature of NC decomposition.

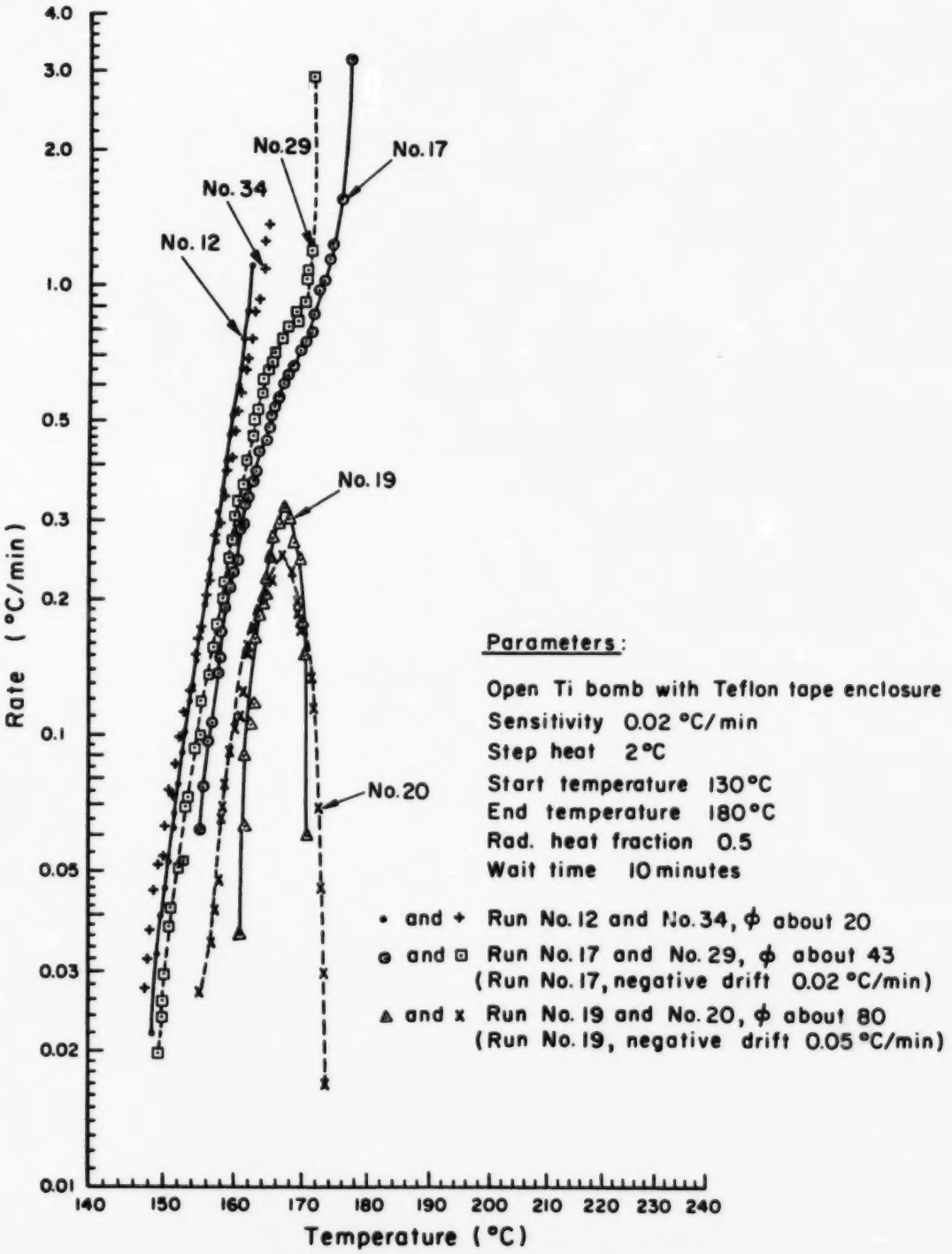

Fig. 4 - Effect of thermal inertia (Ø) and drifting problem on self-heat curve.

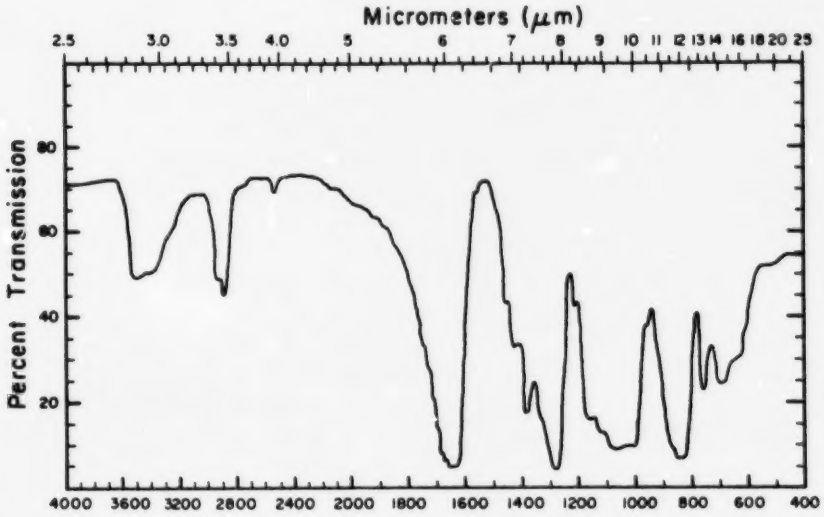

Fig. 5A - Nitrocellulose in desiccator, KBr pellet.

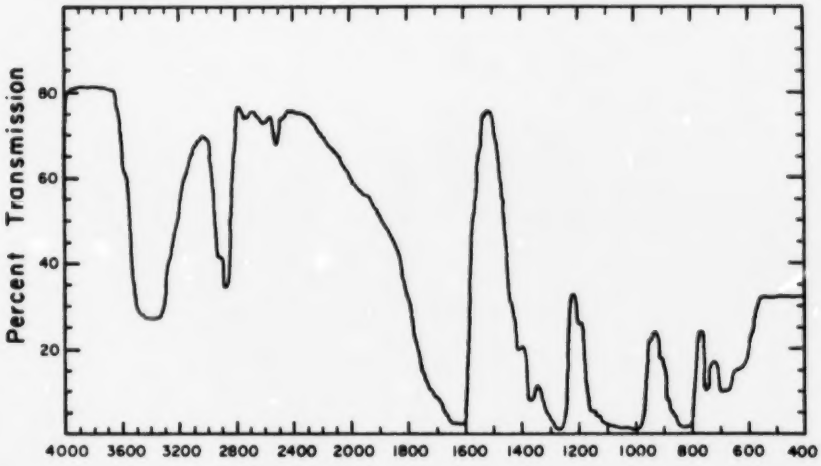

Fig. 5B - Nitrocellulose after heating to self-heat rate of 0.5 °C/min, KBr pellet.

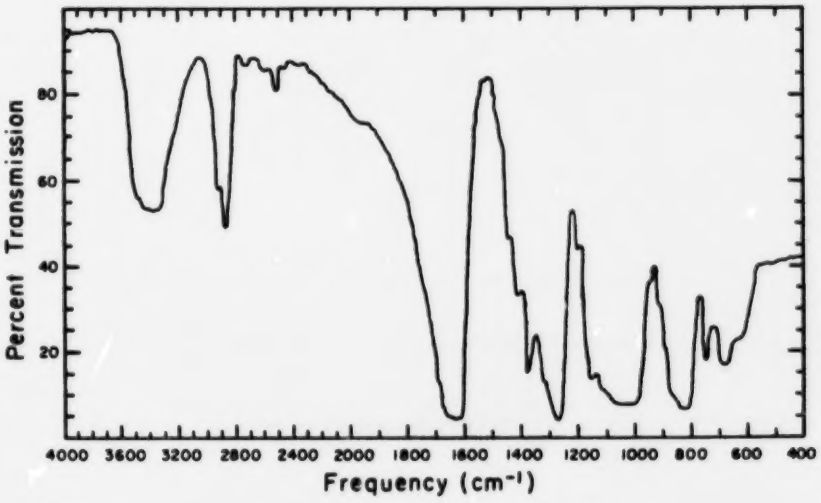

Fig. 5C - Nitrocellulose stored at 75°C for 141 days, KBr pellet.

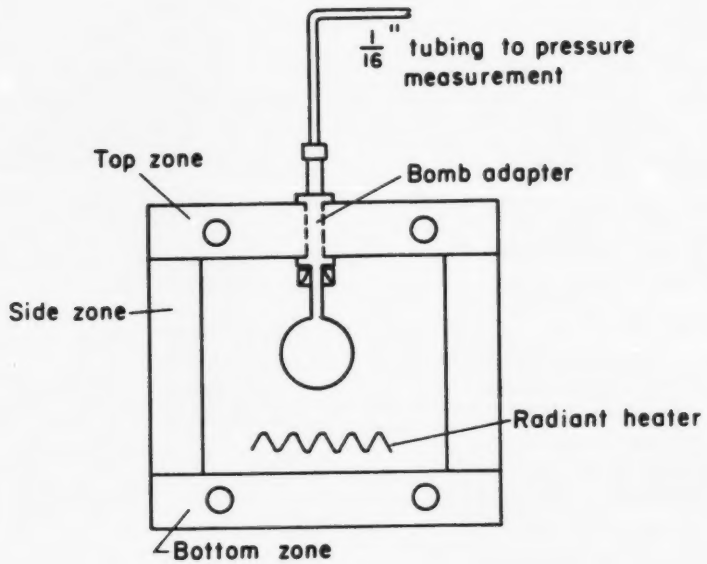

Fig. 6A - Original calorimeter design

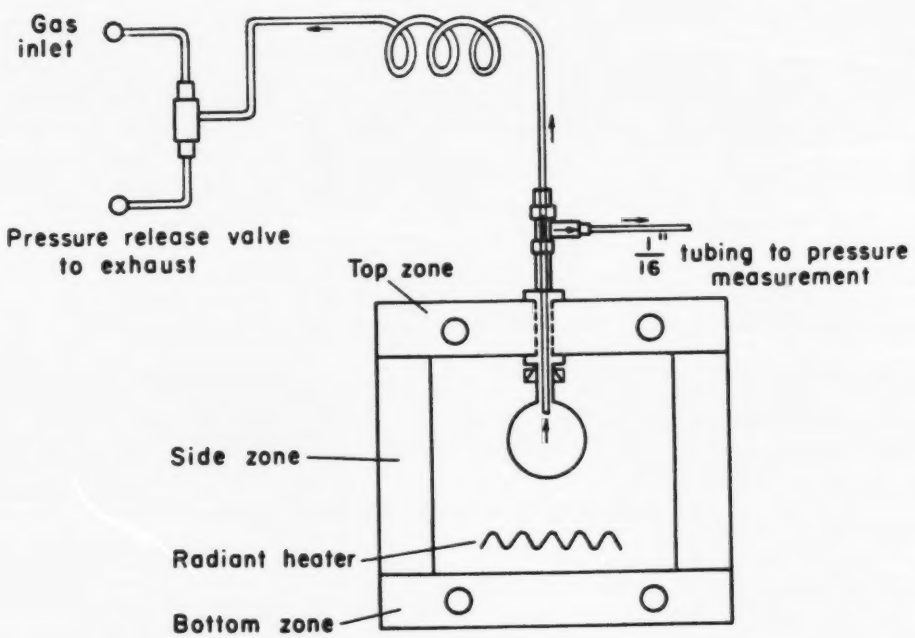

Fig. 6B - Modified calorimeter top design

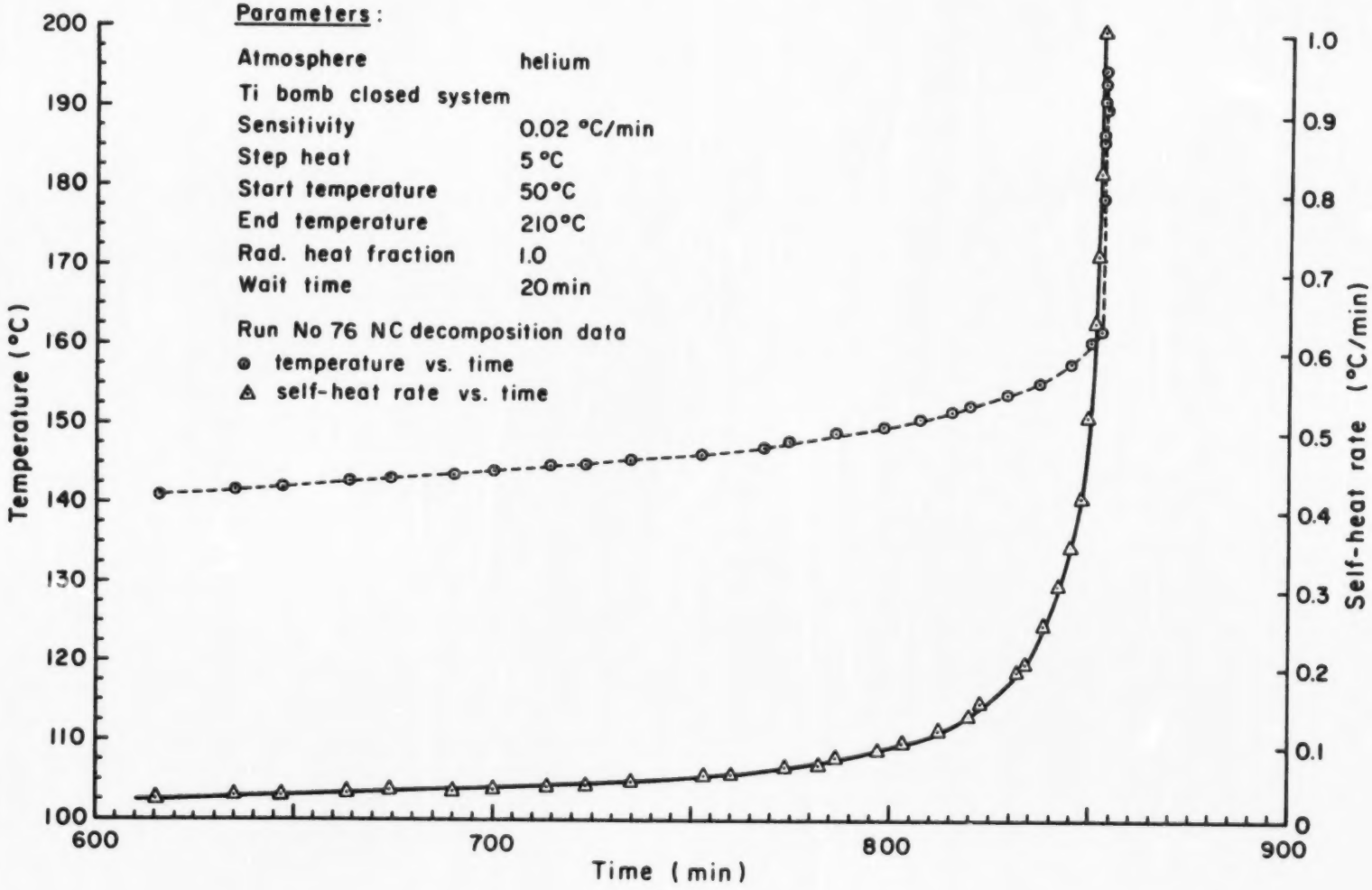

Fig. 7 - Temperature and self-heat rate vs time curves for an adiabatic exothermic decomposition.

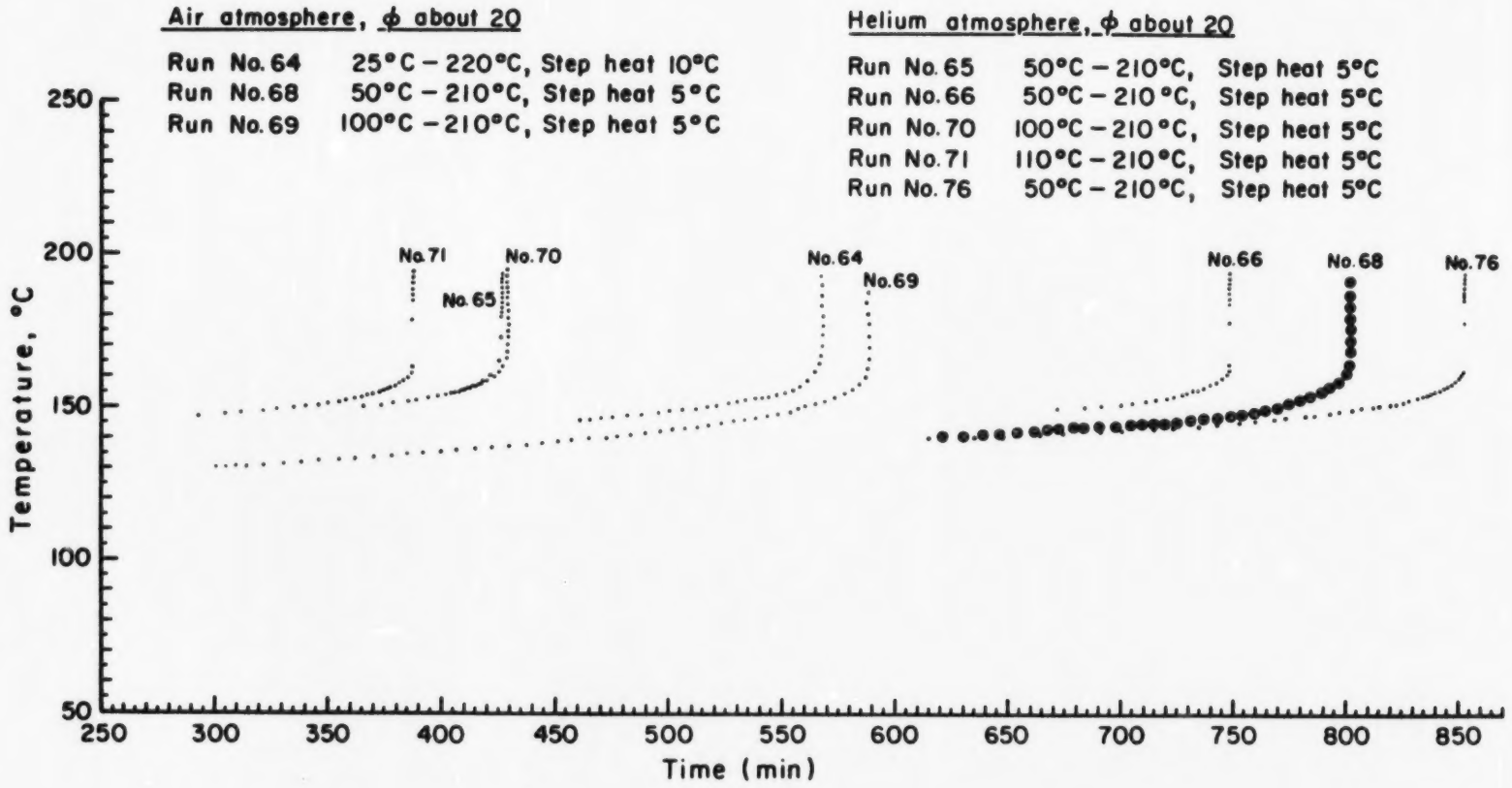

Fig. 8 - NC decomposition in air and helium atmospheres.